

# *New Industrial Technologies and Innovations for the Production of Nanostructured Materials*

Ilya P. Mikhnev

Volgograd Institute of Management - branch of the Russian Presidential Academy of National Economy and Public Administration  
Volgograd, Russia  
mkmco@list.ru

Natalia A. Salnikova

Volgograd Institute of Management - branch of the Russian Presidential Academy of National Economy and Public Administration  
Volgograd, Russia  
ns3112@mail.ru

Svetlana V. Mikhneva

Volgograd Institute of Management - branch of the Russian Presidential Academy of National Economy and Public Administration  
Volgograd, Russia  
svet-mihneva@list.ru

**Abstract**—The development and implementation of nanotechnology is one of the key areas of global technological progress, comparable in value to computer science and communications, or biotechnology. Progress in electronics, medicine, materials science, mechanics, mechanical engineering, and the space industry largely depends on the development of nanotechnologies and the creation of new materials on their basis. The peculiarity of the present moment is that nanotechnologies from the sphere of research have entered the technological market and are the most dynamically developing segment. Research and development on the creation of structural materials based on metals, such as nanostructured steels, alloys, ceramics, etc., are among the priority areas of science and technology in all leading countries of the world. The article discusses the optimal conditions for the synthesis of nanomaterials and the physicochemical fundamentals of nanoindustry technology, describes the characteristics of promising nanostructured materials, methods for producing carbon nanomaterials, the field of application of nanomaterials.

**Keywords**—*nanoindustry; nanosystems; carbon nanostructured materials; nanotechnology; industrial technologies; innovative economy*

## I. INTRODUCTION

The formation of the innovation economy of Russia is predetermined by the dynamics of scientific and technological progress and by the need to develop and implement high-tech technologies in all spheres of the national economy. The leading role in the intensive development of the economy belongs to qualitatively new industrial technologies that ensure a multiple increase in the productivity of equipment and the emergence of materials with new properties and possibilities of their application.

Nanotechnology is a set of knowledge about the ways and means of conducting processes based on the phenomenon of self-organization of nanoscale particles and systems and of the use of the internal capabilities of systems. An analysis of the state and trends in the development of nanoindustry facilities now allows us to conclude that one of the most promising areas of nanotechnology is the synthesis of carbon nanomaterials (SCN) – fullerene-like structures which represent a new allotropic form of carbon in the form of closed, frame, macromolecular systems.

These materials have a number of unique properties due to the ordered structure of their nanofragments: good electrical conductivity and adsorption properties, ability to cold electron emission and gas accumulation, diamagnetic characteristics, chemical and thermal stability, high strength combined with high values of elastic deformation. Nanomaterials can be successfully used as structural modifiers of structural materials, hydrogen accumulators, radioelectronics elements, and additives to lubricants, varnishes and paints, highly effective adsorbents, gas distribution layers of fuel cells [1]. The use of carbon nanostructures in fine chemical synthesis, biology, and medicine is widely discussed [2, 3].

Evaluating the methods of producing SCN from the perspective of industrial production, it should be noted the advantages of catalytic synthesis of SCN in the process of pyrolysis of hydrocarbons [3]. As arguments in favor of this conclusion it should be noted: the relatively low energy intensity of the process; the use of cheap and affordable carbon-containing raw materials; relatively "soft" technological parameters of the synthesis; simplicity of design and manufacturability of the equipment used; no need and expensive purification from impurities [4]. There are various

estimates of the volume of the global market for the production and sale of carbon nanomaterials. According to some estimates the production of SCNs in a few years might increase to several hundred tons and the sales volume will exceed 3 billion euros. The SCN market of domestic production is currently in its infancy, at the same time the leading research organizations of the Russian Federation have obtained significant results in conducting research in the field of carbon nanostructure synthesis.

## II. NANOSTRUCTURED MATERIALS

In the most general terms, nanomaterials can be called any materials whose components (building blocks) are about the size of a nanometer (recall that 1 nm = 10<sup>-9</sup> m). Such components can be large molecules, clusters, particles of a substance or grains of a polycrystal. The route of creating nanoparticles can be represented by the scheme:

atomic vapor → molecules → associates → clusters → nanoparticles and nanocomposites.

The concept of a nanomaterial must include an abrupt change in the properties of a substance, which is observed when the nanometer size of the blocks constituting the nanomaterial is reached. The threshold particle size, which determines the stepwise change in the properties of a substance – the size effect – for most materials currently known, ranges from 1 to 100 nm. The nanoeffect is caused by a jump-like change in the activation energy (E<sub>a</sub>) of the process, regardless of whether it proceeds in the kinetic, diffusion, or mixed modes.

Micrometer, like nanometer, is a quantitative characteristic of particle size. But between the nanostate and submicrostate there is a fundamental difference. It is the nano- and not submicrostate that is intermediate between the molecular and solid state, in which the cooperative effects are manifested. This is also the physical reason for the sudden change in properties observed in the nanometer range. The distances at which physical forces manifest themselves vary in the range from 1 to 100 nm. In this connection, they can manifest themselves in a particular substance at different sizes of nanoparticles [5].

Consequently, there can not be a fundamental size of nanoparticles, both for the same and for different substances. Nanomaterials are not always crystalline particles. As a rule, they are thermodynamically nonequilibrium systems, and therefore the particles forming them do not necessarily have a crystal-perfect structure. On the contrary, the building blocks of nanomaterials are often characterized by a highly defective structure, sometimes their state is close to amorphous. In other words, in nanoparticles, the long-range order can be strongly disturbed, and the correlation of the cooperative effect is determined by the short-range order. In this regard, a more accurate name of nanomaterials – nanostructured materials.

## III. OPTIMAL CONDITIONS FOR THE SYNTHESIS OF NANOMATERIALS

For optimal organization of the working area of equipment for the production of nanomaterials, the following should be considered:

- the certainty and reproducibility of the initial state of a set of reagents is achieved the more difficult the more complex the system (phase and chemical composition, the activity of the components, their history);
- the degree of disequilibrium of the transition processes from the initial to the final products is determined by the driving force of the process, which determines the reaction mechanism, which has a complex effect on the nature of the final product;
- the duration of the path (staging, speed) from the initial to the final products determines the determination in the behavior of the system. It should be borne in mind that increasing the temperature of the process increases the number of routes and intermediate products. It is advisable to conduct impulse effects lasting a millisecond, leading to the activation of the reaction mass (thermodynamic processes). Unfortunately, today only an empirical approach allows you to optimize the conditions of impact on the system;
- whenever possible, elimination of bifurcations of the process in the reaction zone, in particular, the use of seeds in the form of perfect crystals of the initial product;
- use of computer simulation to find ways to eliminate chaos.

## IV. PHYSICO-CHEMICAL BASES OF NANOINDUSTRY TECHNOLOGY

At present, a large number of methods for producing nanoparticles have been developed, which make it possible to very finely regulate the size of particles, their shape and structure. They can be divided into three groups: physical, chemical and mechanochemical methods (Fig. 1).

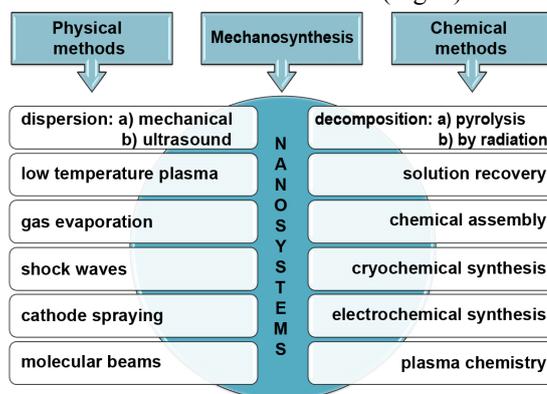


Fig. 1. Ways to get nanosystems.

For physical methods, a characteristic feature is the production of particles by dispersion, the so-called “top” approach, for chemical methods, the production of particles by enlarging individual atoms, or the “bottom” approach. It is fundamentally important that the structure of the particles of the same size obtained in this case may differ. When dispersing to nanoparticles, as a rule, the structure of the initial compact material is preserved, while aggregation, the resulting particles may have a different spatial arrangement.

Grinding (dispersion) of materials by mechanical means in mills of various types was widely used even before the era of nanotechnology. It should be borne in mind that when crushing to large particles, the energy consumption is proportional to the volume of the body being destroyed, and when producing nanoparticles, the work of grinding is proportional mainly to the area of the resulting surface. Therefore, in this case, the use of high-power mills - attritors and simoloyers. These are high-energy grinding devices with a fixed body - a drum and agitators, transmitting movement to the balls in the drum. The rotation speed of the mixers can reach 3000 rpm. Attritors have a vertical arrangement of the drum, simoloyera - horizontal. The grinding of material by grinding balls, unlike other types of grinding devices, occurs mainly not due to impact, but by the mechanism of abrasion. The capacity of the drums in the installations of these two types reaches 400 - 600 liters.

Mechanically crushed metals, ceramics, polymers, oxides, brittle materials. The degree of grinding depends on the type of material. So, for tungsten and molybdenum oxides, particles of about 5 nm are obtained, for iron - about 10 - 20 nm. The positive side of mechanical methods of grinding is the comparative simplicity of installations and technologies, the ability to grind various materials and obtain powders of alloys, as well as the ability to obtain material in large quantities. The disadvantages of the method include the possibility of contamination of the powder being ground with abrasive materials, as well as the difficulties of obtaining powders with particles of the same size and controlling the composition of the product during the grinding process.

Another common mechanical method for producing nanoparticle powders is dispersion of melts by a stream of liquid or gas. This is a high-performance process that is easy to implement on a continuous basis and automate, it is economical and environmentally friendly. Powders of metals and alloys of Fe, Al, Cu, Pb, Zn, Ti, W, etc. are obtained by this method. This method can be used for large-scale production of nanostructured powders. The obtained nanostructured powder can be used to obtain bulk samples. The most common production methods are powder technology, i.e. various types of pressing and sintering.

In the molecular beam method, liquid or solids are evaporated at a controlled temperature in a low-pressure inert gas atmosphere, followed by condensation of steam in a cooling medium or on cooling devices. This method allows to obtain particles ranging in size from two to several hundred nanometers. Molecular beams obtained by slow (effusion) outflow of evaporating particles have a low intensity. The temperature of the source is chosen depending on the required

intensity of the molecular beam and the equilibrium pressure over the evaporated material. It may be higher or lower than the melting point of the substance. It should be noted that some substances (for example, Sn and Ge) evaporate both as individual atoms and as small clusters. In molecular beams of low intensity, obtained by effusion through a hole in the heating chamber, a uniform distribution of clusters of small sizes is observed. The main advantage of the molecular beam method is the ability to quite accurately control the intensity of the beam and control the feed rate of particles into the condensation zone. For gas-phase production of nanoparticles, installations are used that differ in the methods of supplying and heating the evaporated material, the composition of the gaseous medium, the methods of performing the condensation process and the selection of the resulting powder. For example, the powder is precipitated onto a cooled rotating cylinder or drum and is scraped from it with a scraper into the receiving container. Unlike evaporation in a vacuum, the atoms of a substance evaporated in a rarefied atmosphere lose kinetic energy more quickly due to collisions with gas atoms and form nuclei of crystals (clusters). When they condense, nanocrystalline particles are formed. So, in the process of condensation of aluminum vapors in the environment of hydrogen, helium and argon at different pressures of gases, particles of 20-100 nm are obtained. Shock wave or detonation synthesis [8] is most effective for materials that are formed at high pressures, for example, powders of diamond, cubic boron nitride (elbor), and others. Depending on the power and type of the explosive device, the shock-wave interaction on the material is carried out in a very short period of time (tenths of microseconds) at a temperature of over 3000 K and a pressure of several tens of gigapascals. Under such conditions, a phase transition in substances is possible with the formation of ordered dissipative nanoscale structures.

In the explosive conversion of condensed explosives with a negative oxygen balance (a mixture of TNT and RDX), carbon is present in the reaction products, from which the dispersed diamond phase is formed with a particle size of about 4 to 5 nm. By subjecting the explosives to shock-wave action of the porous structures of various metals and their salts, gels of metal hydroxides, one can obtain nanopowders of Al, Mg, Ti, Zn, Si oxides and others. The advantage of the shock-wave synthesis method is the possibility of obtaining nanopowders of various compounds not only of ordinary phases, but also of high-pressure phases. However, the practical application of the method requires special facilities and process equipment for blasting. Electrochemical synthesis is associated with the release of simple and complex cations and anions at the cathode of a substance during electrolysis.

If a system consisting of two electrodes and a solution (melt) of an electrolyte is included in the DC circuit, then oxidation-reduction reactions will take place on the electrodes. At the anode (positive electrode), anions donate electrons and oxidize; at the cathode (negative electrode) cations attach electrons and are restored. The precipitate formed on the cathode as a result of, for example, electrocrystallization, may be morphologically both a loose and dense layer of a variety of microcrystallites. The texture of the precipitate is influenced by many factors, such as the

nature of the substance and solvent, the type and concentration of ions of the target product and impurities, adhesion properties of the deposited particles, medium temperature, electric potential, diffusion conditions, and others.

One of the promising scientific directions is the use of electrochemical synthesis for the design of nanostructured materials. Its essence lies in the formation during the electroreduction of metal nanoparticles under the layers of organic, including polymeric, compounds. The main advantages of the method are the experimental availability and the ability to control and control the process of obtaining nanoparticles. One of the most common chemical methods for producing ultrafine powders of metals, nitrides, carbides, oxides, borides, as well as their mixtures is plasma-chemical synthesis. This method is characterized by a very fast ( $10^{-3}$ – $10^{-6}$  s) reaction proceeding far from equilibrium and a high rate of formation of new nuclei at a relatively low rate of their growth. Plasma-chemical synthesis uses a low-temperature (4000 – 8000 K) nitrogen, ammonia, hydrocarbon, argon plasma, which is created using an electric arc, an electromagnetic high-frequency field, or a combination of them in reactors called plasmatrons. In them the stream of initial substances (gaseous, liquid or solid) quickly flies through the zone where the plasma is supported, receiving energy from it for carrying out chemical transformation reactions. The plasma-forming gas can also be the starting material itself.

The processes occurring during plasma-chemical synthesis and the gas-phase method for producing nanoparticles are close to each other. After the interaction, the formation of active particles in the gas phase occurs in the plasma. In the future, it is necessary to preserve their nano-dimensions and isolate them from the gas phase. Powders of plasma chemical synthesis are characterized by a wide distribution of nanoparticles in size and, as a result, the presence of rather large (1 – 5  $\mu\text{m}$ ) particles, that is, low selectivity of the process, as well as a high content of impurities in the powder. In the processes of thermal decomposition, complex organometallic compounds, hydroxides, carbonyls, formates, nitrates, oxalates, and metal amides are usually used, which decompose at a certain temperature to form the synthesized substance and release the gas phase. For example, pyrolysis of formates of iron, cobalt, nickel, and copper in vacuum or inert gas at a temperature of 470 – 530 K produces dispersed powders of metals with an average particle size of 100 – 300 nm. Nanocrystalline powder of aluminum nitride (AlN) with an average particle size of 8 nm was obtained by decomposition of aluminum polyamide in ammonia at 900 K. Borides of transition metals can be obtained by pyrolysis of borohydrides at 600 – 700 K, that is, at a temperature that is much lower than ordinary solid-phase synthesis temperatures.

It should be noted that during the pyrolysis of Cu and Ni formates, the yield of free metal predominates, and during the pyrolysis of Mn and Fe formates, the yield of metal oxides prevails. Other metal formates may be intermediate; for example, during the pyrolysis of cobalt formate, 50-60% CoO and 50 – 40% Co are formed.

In cryochemical synthesis, a solution containing cations of the synthesized material undergoes rapid freezing and freeze-drying in vacuum, followed by thermal decomposition. The product of the synthesis is usually an oxide powder with a crystallite size of 40 – 300 nm, the degree of agglomeration of which essentially depends on the choice of the substance to be frozen (solution / suspension / precipitate). It is also possible to remove ice by low-temperature extraction in polar organic solvents (cryoextraction).

When mechanosynthesis provide mechanical processing of solids, as a result of which the grinding and plastic deformation of substances occur. The grinding of materials is accompanied by breaking of chemical bonds, which predetermines the possibility of the subsequent formation of new bonds, that is, the flow of mechanochemical reactions. Mechanical impact when grinding materials is pulsed; however, the occurrence of the stress field and its subsequent relaxation do not occur during the entire residence time of the particles in the reactor, but only at the time of the collision of the particles and in a short time after it. In addition, the mechanical effect is local, since it occurs not in the whole mass of solid matter, but only where the stress field arises and then relaxes. The impact of energy released at a high degree of disequilibrium during impact or abrasion, due to the low thermal conductivity of solids, leads to the fact that some part of the substance is in the form of ions and electrons – in the plasma state.

The mechanochemical processes in solids can be explained using the phonon theory of the destruction of brittle bodies (phonon – quantum of energy of elastic oscillations of the crystal lattice). Mechanical grinding of solid materials is carried out in the mills of ultrafine grinding (ball, planetary, vibration, jet). When the working bodies interact with the material being crushed, its local short-term heating to high (plasma) temperatures is possible. Mechanically, nanopowders can be obtained with a particle size of from 5 – 10 to 200 nm. So, when grinding a mixture of metal and carbon for 48 hours, particles of TiC, ZrC, VC and NbC with a size of 7 – 10 nm were obtained. In a ball mill, particles of a WC – Co nanocomposite with a particle size of 11 – 12 nm were obtained from a mixture of tungsten, carbon, and cobalt powders with an initial particle size of about 75 microns for 100 hours.

## V. METHODS FOR PRODUCING CARBON NANOMATERIALS

Despite the fact that by now already dozens, if not hundreds, of research organizations around the world have the equipment for the synthesis of SCN, they all use a technique that implements three main ways:

- arc;
- laser ablation;
- synthesis of CNM from carbon-containing gases.

### A. Arc Method

The most widely used method is to obtain SCN using thermal sputtering of a graphite electrode in an arc discharge plasma burning in a helium (He) atmosphere. The method used in 1991 by the Japanese scientist S. Iijima differed from the method of producing fullerenes in that the electrodes were not in contact with each other, but were located at some distance from each other during the arc. Under these conditions, carbon evaporating from the anode condenses on the cathode in the form of sediment of predominantly cylindrical form. Were obtained carbon nanotubes in the form of sharp needles with a diameter of 4 to 30 nm and a length of 1  $\mu\text{m}$  at the negative end of the carbon electrode at a constant arc current discharge. The modification of the method, which consisted in finding the optimal parameters: He pressure, the magnitude of the arc current, voltage, and the gap between the electrodes, made it possible to increase the yield of nanotubes.

In the arc discharge between the anode and the cathode at a voltage of 20 – 25 V, a stabilized direct arc current of 50 – 100 A, an interelectrode distance of 0.5 – 2 mm and a pressure of 100 – 500 Torr, an intensive sputtering of the anode material occurs. A part of the sputtering products containing graphite, carbon black and fullerenes is deposited on the cooled walls of the chamber, a part containing graphite and multi-layered carbon nanotubes is deposited on the cathode surface. A distinctive feature of this method of synthesizing SCN is that it is with its help that the most qualitative SCNs are obtained, up to several micrometers in length with similar morphological parameters and small diameter (1 – 5 nm).

However, it should be noted that the achievement of such high quality is associated with great technological difficulties associated primarily with the need to implement a multi-stage purification of the product from soot inclusions and other impurities. The output of the SCN does not exceed 20 – 40%. Many factors influence the stability of the technological process, and, consequently, the quality of the SCN. These are voltage, force and current density, plasma temperature, total pressure in the system, properties and feed rate of inert gas, dimensions of the reaction chamber, synthesis time, presence and geometry of cooling devices, nature and purity of the electrode material, their ratio of geometric dimensions, and a number of parameters that are difficult to quantify, for example, the cooling rate of carbon vapor, etc. Such a huge number of control parameters complicates the process control, instrumentation design of synthesis plants and puts an obstacle to their reproduction in the scale of industrial application.

### B. Graphite Laser Evaporation

In 1995, a group of R. Smolireported on the synthesis of SCN by laser evaporation (ablation). A pulsed or continuous laser was used to vaporize a graphite target in a furnace heated to 1200 ° C. The chamber in the furnace was filled with helium or argon with a pressure in the range of 500 Torr. In the course of evaporation, a very hot cloud of vapor was formed, which then stretched and cooled rapidly. Molecules and carbon atoms condensed to form large

molecules, including fullerenes. The catalysts also began to condense, but more slowly, and, joining carbon molecules, prevented their closure. SCNs were formed from these initial carbon molecules until the catalyst particles became too large or until they cooled enough so that carbon could no longer diffuse through or across the surface of the catalyst particles. It is also possible that the catalyst particles were covered with a layer of amorphous carbon and could no longer adsorb it, and the growth of the SCN stopped.

In the method under consideration, compared with the arc, the number of parameters determining the performance and morphology of the SCN is much smaller. Therefore, the prospect of this method of synthesizing SCN as an industrial application object seems more realistic. However, it should be noted that the implementation of laser synthesis involves the use of very expensive and difficult to operate equipment, requires a large amount of energy expended. The formation of carbon vapor occurs at 3000 ° C from the solid phase (target) in a highly non-equilibrium state. The nanotubes thus formed are mixed with the target material, which makes it difficult to clean and, therefore, make practical use of the material obtained.

### C. Synthesis of Carbon Nanomaterials From Carbon-Containing Gases

According to the initial raw materials, two groups of processes can be distinguished, the first of which includes the disproportionation of CO, the second – the pyrolysis of hydrocarbons. The works of R. Smalley laid the foundation for the creation of the HiPCO process (The High Pressure CO) – methods for catalytic production of CNM in a continuous stream of CO (feedstock) using Fe (CO)<sub>5</sub> as an iron catalyst. Nanotubes are obtained by passing CO mixed with Fe (CO)<sub>5</sub> through a heated reactor.

This method produced nanotubes with a diameter of only 0.7 nm, which are assumed to have the smallest sizes of achievable chemically stable CNMs. The average diameter of the obtained CNMs in the HiPCO process is approximately 1.1 nm. The second group of synthesis processes (pyrolysis) from coal-containing gases includes much more options. In principle, any carbon-containing substances can undergo pyrolysis. It follows from the above that almost any carbon-containing gases can be used as a carbon source for the synthesis of SCN. However, when creating a technology for industrial synthesis of SCN, it is advisable to choose the most affordable and cheap gases, besides providing high performance, for example, methane or propane-butane mixtures.

## VI. APPLICATIONS OF NANOMATERIALS

The Russian Academy of Sciences based on fundamental research provides for obtaining new knowledge about the properties of nanostructures and nanomaterials, developing the fundamental principles of nanotechnology, including self-assembly processes and creating nanomechanisms and nanosystems, developing methods and technical means for studying and manipulating nanoscale objects, while technologies that will make a significant

contribution to solving social issues. In the study of nanomaterials, the focus is on high-performance coatings and materials to create new products and production processes. Among the promising new materials are nanocomposites, biomaterials and hybrid materials consisting of organic and inorganic elements. The importance of creating advanced tools for monitoring production processes and studying the characteristics of the materials obtained, as well as the importance of developing methods for mathematical modeling of nanostructures, is noted.

The results of research and development in the field of nanotechnology should lead to the creation of new industrial technologies and industries to ensure such areas as construction, transport, energy, environmental protection, computer science and communications, healthcare, food production, etc. [6, 7]. The development of nanotechnology of the Russian Academy of Sciences identified the following priority areas: carbon nanomaterials; new materials and technologies for nanoelectronics, spintronics optoelectronics; organic and hybrid nanomaterials; polymers and elastomers; crystalline materials with special properties; mechatronics and microsystem technology; composite and ceramic materials; membranes and catalytic systems; biocompatible materials; nanodiagnostics and probe methods [8, 9].

The Federal target program for the development of nanoindustry of the Russian Federation until 2020 provides for the following thematic areas: nanoelectronics; nanoengineering; functional nanomaterials and high-purity substances; functional nanomaterials for energy; functional nanomaterials for space technology; nanobiotechnology; structural nanomaterials; composite nanomaterials; nanotechnology for security systems [10]. Integral indicators of the development of large areas in nanotechnology and the relationship between them can serve as data on patents obtained (Figure 2) and on the structure of the global market for nanotechnology products (Figure 3).

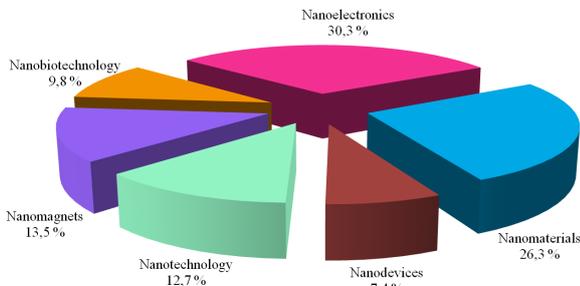


Fig. 2. The distribution of patents in the field of nanotechnology in 2017.

According to the global nanotechnology market structure presented in Figure 3, in 2017, the total volume of the nanotechnology products market reached 2.51 trillion dollars.

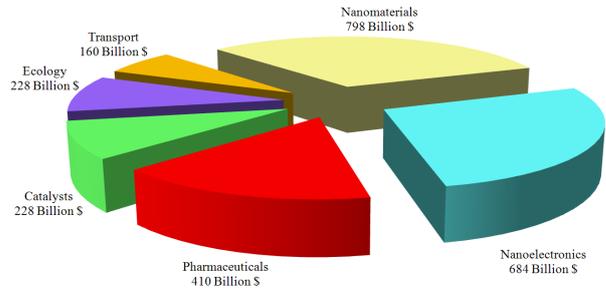


Fig. 3. The structure of the global nanotechnology market in 2017 (figures indicate the volume of market sectors in US dollars).

## VII. CONCLUSION

It should be emphasized that the development of the science of nanostructures and quantum nanostructures (nanophysics) and nanotechnologies will enable the production of nanomaterials with qualitatively new properties [11]. The development of nanoelectronics and nanomechanics will serve as a basis for a qualitatively new stage in the development of new information technologies, means of communication. It will solve the problems of a qualitatively new standard of living, etc. Success in developing these areas will be determined by solving two main problems: developing reliable ways to create nanomaterials and nano objects with the required properties, including the use of methods of atomic assembly and the effects of self-organization; development of new and development of existing methods of nanodiagnostics with atomic resolution. Modern progress in the field of nanotechnology gives hope that in the near future many problems will be solved.

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